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Modern Nondestructive Test Methods for Army Ceramic Matrix Composites

by Douglas J. Strand

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14. ABSTRACT
As composites have developed and matured, the nondestructive testing (NDT) methods needed to characterize and maintain them have also developed and matured. The purpose of this report is to examine the newer methods of NDT, which may be applicable to some of the new composite materials being used for U.S. Army applications. Ceramic matrix composites (CMC) are potentially good high-temperature structural materials because of their low density, high elastic moduli, high strength, and for those with weak interfaces, surprisingly good damage tolerance.

Ultrasonic testing (UT) has long been one of the most widely used and most effective methods of inspecting composite materials. Some new types of UT are the ultrasonic multiple-gate C-scan technique, resonant ultrasound spectroscopy, phase UT, acousto-ultrasonics, liquid crystal display UT, and flexible transducer UT. In addition to UT, several other nondestructive test techniques have recently been found to be effective with some of these new composite materials. They include transient thermal conductivity measurements, flexural wave with holography NDT, pulsed thermography, and the dynamic characterization technique.

The report will describe each of these new techniques, give the advantages and disadvantages of each one, state when and with what materials and systems each method is usable, and combine finite-element analysis with conventional analysis of nondestructive methods for examining CMCs.

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1. Introduction

As composites have developed and matured, the nondestructive testing (NDT) methods needed to characterize and maintain them have also developed and matured. The purpose of this report is to examine the newer methods of NDT, which may be applicable to some of the new composite materials being used for U.S. Army applications. Ceramic matrix composites (CMC) are potentially good high-temperature structural materials because of their low density, high elastic moduli, high strength, and for those with weak interfaces, surprisingly good damage tolerance (*1*).

Ultrasonic testing (UT) has long been one of the most widely used and most effective methods of inspecting composite materials. Some new types of UT are the ultrasonic multiple-gate C-scan technique, resonant ultrasound spectroscopy, phase UT, acousto-ultrasonics, liquid crystal display UT, and flexible transducer UT. In addition to UT, several other nondestructive test techniques have recently been found to be effective with the Army's new composite materials. They include transient thermal conductivity measurements, flexural wave with holography NDT, pulsed thermography, and the dynamic characterization technique.

The report will describe each of these new techniques, give the advantages and disadvantages of each one, state when and with what materials and systems each method is usable, and combine finite-element analysis with conventional analysis of nondestructive methods for examining CMCs.

2. Modern Ultrasonic Test Methods for CMCs

The ultrasonic multiple-gate C-scan technique, also known as "software gating," features multiple peak-detection gates between the front and back surface echoes on the A-scan signal. Since each gate records the amplitude variation for a very narrow time-of-flight range, the frequent fluctuations in signal amplitude due to the inhomogeneity of the material affects one or two gates at times, while the other gates remain sensitive to small amplitude signals from defects. This increased sensitivity allows the detection of very small material defects such as porosity. Optical microscopy can then be used to show more extensive porosity and regions of poor consolidation in matrix material at the depth indicated by the C-scan.

This technique gives very good near-surface resolution of delaminations and provides high resolution in separating adjacent-ply delaminations. This high resolution is achieved by using multiple peak-detection gates placed between the front and back ultrasonic surface echoes from within the composite, together with a software-based, front surface tracking algorithm. This ability to single out defect echoes as a function of specimen depth (or ultrasonic time-of-flight), while minimizing interference from material noise, makes this technique attractive for use in detecting porosity in CMCs (*2*).

The resonant ultrasound spectroscopy (RUS) technique is based on ultrasonic excitation and the measurement of the mechanical resonant frequencies of a small sample having a regular shape. The mechanical resonant response of a solid is a function of its elastic moduli, shape, and density. The resonant spectrum can be determined based on these parameters. However, no analytical solution exists for the inverse problem, i.e., determining the elastic constants from a measured spectrum. Therefore, to deduce the elastic constants by the RUS technique, a least-square modeling approach is used. The resonant frequency spectrum is first calculated from an initial estimate for each of the elastic constants. This calculated spectrum is then compared with the measured one, and a least-square difference between the two is calculated and summed for all the spectral peaks to find a residual parameter, *F*, given by $F = \sum_i w_i (f_i^* - f_i)^2$, where f_i (i = 1,2,…,n) is the ith measured resonant frequency, f_i^* the predicted frequency, and w_i a weight factor reflecting the degree of confidence in the measured frequency. The minimum value of *F* is then found by varying the estimated elastic constants in the forward problem that predicts f_i^*. The resulting elastic constants are then considered to be the actual elastic constants of the material. This method has already been used successfully to determine the complete elastic constants of many single-crystal materials, intermetallic compounds, and recently, boron fiber-reinforced aluminum composites. It is necessary to choose a symmetry for the RUS method. Transversely isotropic symmetry, orthotropic symmetry, and tetragonal symmetry have been used successfully with CMCs. One result obtained using this method with unidirectional and 0°/90° crossply Nicalon*-SiC fiber-reinforced calcium aluminosilicate (CAS) CMCs is that the overall elastic anisotropy is small. This result is due to the fact that the modulus ratio of the Nicalon-SiC fiber to the CAS matrix is relatively low and the fiber volume fraction is moderate (*1*).

Phase UT is also called the ultrasonic harmonic wave method. The application of this method for testing of composite materials with irregular structures as well as for joints of materials with distinctive properties may allow characterization of a wider class of materials, including CMCs. Stochastic approaches are used to analyze distribution of mechanical properties for characterization of materials. It is proposed to use this method for the monitoring of multilayered composite materials using the physical process of harmonic wave diffraction of the layers with different acoustic properties (*3*).

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*Nicalon is a registered trademark of Nippon Carbon Company Ltd., Tokyo, Japan.

Acousto-ultrasonics (AU) is a through transmission or pitch-catch method whose purpose is to correlate certain empirical parameters of the detected waveform to characteristics of the material between the two transducers. There are basically two approaches for analyzing the received diffuse, i.e., multimode AU signal. The diffuse field decay rate method, used extensively at NASA Glenn Research Center, involves quantifying the internal damping of vibrational energy in materials. The decay rate is determined by dividing the recorded waveform into a number of time windows and then performing fast Fourier transforms (FFT) on each of the time windows to obtain the power spectra. The total energy of each time window is calculated using the respective power spectrum, i.e., the area under the power spectrum plot.

The second method for analyzing the AU signal involves working with the entire time domain signal and again conducting an FFT in order to obtain the power spectrum. Certain parameters, called shape parameters, were shown to be sensitive to various types of damage in composite materials. The critical shape parameter is the attenuation due to internal damping for which information is being collected from the frequency domain. The three shape parameters used to indirectly quantify the attenuation are the ultrasonic decay rate, the mean square value of the power spectrum, and the centroid of the power spectrum. Values for these three parameters were experimentally found in two types of SiC/SiC CMC that contained damage sites. Both systems contained Hi-Nicalon fibers with a carbon interface but had different matrix compositions that led to considerable differences in damage accumulation. In addition to correlating well with predicted damage mechanisms, AU results were shown to be dependent upon the stress states of the composites. This stress-dependent behavior was seen while unloading specimens from the maximum stress, thereby maintaining a constant damage state (*4*).

Liquid crystal display UT is also called acoustography. It is a full-field ultrasonic imaging process in which a high-resolution, two-dimensional acousto-optic sensor based on liquid crystal technology is employed to directly convert the ultrasound into a visual image in near real time. A series of images develops as a function of time in the acoustography process. Data seen in early frames is sometimes lost in later frames once steady state has been achieved, making quantitative determination of discontinuity size and relative attenuation differences between regions hard to distinguish from each other. Processing techniques have been developed that allow the reduction of an acoustographic image sequence to a single, easily interpreted image that improves discontinuity detection, increasing the dynamic range of the technique. Reduction to a single image allows an easier determination of relative attenuation and discontinuity size. This technique is promising for polymer and ceramic matrix composites (*5*).

Flexible ultrasonic transducers (FUT) consist of a metal foil, a piezoelectric ceramic film, and a top electrode. The flexibility is realized due to the porosity of the piezoelectric film and the thinness of metal foil. It is common to use stainless steel (SS), lead-zirconate-titanate (PZT/PZT) composite, and silver paste for the metal foil, piezoelectric film, and top electrode materials, respectively. The SS foil serves as both substrate and bottom electrode. The PZT/PZT piezoelectric composite film is made by the solgel spray technique. In experimental work with this transducer on a graphite/epoxy composite plate, the center frequency and 6-dB bandwidth were about 750 and 880 kHz, respectively. The low center frequency and bandwidth are the result of the high ultrasonic attenuation within the thick composite plate. It is hypothesized that the signal-to-noise ratio can be improved significantly if the thickness of the PZT/PZT film increases and the center frequency of the flexible FUT decreases (*6*).

3. Other Modern NDT Methods for CMCs

Transient thermal conductivity measurements are of two categories—transient and steady state. Both categories create a temperature gradient and then monitor the response of the material to the gradient. Some of the methods are destructive, such as the laser flash diffusivity technique, and some are nondestructive, such as the modified hot wire and transient plane source techniques. All three of these are of the transient category. These latter two techniques are nondestructive since they are reflectance methods that operate by applying and measuring heat at the same surface. Steady-state techniques, such as the guarded hot plate described in American Society for Testing Materials (ASTM) standard C518 (*7*) from 1985 or the method described in ASTM D5470-93 (*8*) from 1993 involve placing a solid sample of fixed dimension between two temperature-controlled plates. One plate is heated while the other is cooled, and the temperatures of the plates are monitored until they are constant. The thermal conductivity is calculated using the thickness of the sample, the steady-state temperatures, and the heat input per unit time to the hot plate using the equation $Q = \frac{kA}{L}\left(T_{hot} - T_{cold}\right)$, where Q is heat, k is thermal conductivity, A is the cross-sectional area, L is the travel distance from the hot to the cold side of the solid material, and T is temperature. The disadvantages of the steady state techniques are that the sample must conform to specified dimensions, the measurement requires access to both sides of the specimen, and the test time is lengthy in order to allow the steady state to be reached (*9*).

The flexural wave and holography technique uses a piezoelectric transducer attached to a specimen composite ceramic-metal plate to generate transient flexural waves and transmit them through the plate. These pulses are a narrow band signal generated in the time domain. The characteristic wavelengths corresponding to the narrow frequency spectrum may be larger than the size of the flaw. The response is measured by dynamic holographic interferometry using a double-pulsed, twin-cavity Nd:YAG laser, made up of two independent reference beams. The main feature of composite plates is the plates' displacement field (u,v,w) has to be derived on a neutral plane whose in-plane displacements vanish. The experimental interferogram phase maps coincide rather well with analytic results derived from Mindlin's plate equations, including shear and rotary inertia effects. Mindlin introduces a constant k in the stress equations for the shear forces in terms of the shear strain. He obtained the value of *k* from the limiting case of very large frequencies, leading to Rayleigh waves and corresponding wave speeds. *K* depends only on Poisson's ratio, *v*. The stress equations of motion for three-dimensional (3-D) elasticity are integrated over the thickness of the plate, which leads to a set of three differential equations. A fourth differential equation of motion is obtained by eliminating all terms containing Ψ_x and Ψ_y, factors of the displacement field components. Finally, with the principle of superposition and both Fourier analysis and synthesis, the wave field due to any force, not only harmonically oscillating forces applied on any contact surface, but point sources between transducer and plate, can be evaluated (*10*).

Pulsed thermography involves heating a specimen with a short duration pulse of energy and then monitoring the transient thermal response of the surface of the specimen with an infrared camera. The thermal energy on the surface conducts into the cooler interior of the specimen. At the same time, there is a reduction of the surface temperature over time. This surface cooling will take place in a uniform manner as long as the material properties are consistent throughout the sample. Subsurface discontinuities that possess different material properties, such as thermal conductivity, density, or heat capacity, will affect the flow of heat in that particular region. This resistance in the conductive path causes a different cooling rate at the surface directly above the discontinuity than at the surface above a volume, free of discontinuities. The change in the subsurface conduction is seen as a nonuniform surface temperature profile which is expressed as function of time. Discontinuities that are located at greater depths will show up later in time since the technique depends on the interaction of the discontinuity with the advancing thermal front. Deeper discontinuities will tend to have less contrast than near surface discontinuities because of lateral diffusion. Thus, the critical discontinuity size capability of a thermographic test system depends on the discontinuity size, the depth, and the material properties of the component being tested (*11*).

One advantage of pulsed thermography is its large field of application, while one disadvantage is the processing and interpretation of the acquired thermal image data. Three methods used to examine such data for a composite plate with material inserts that simulate delamination type discontinuities are peak contrast, peak slope, and a newly developed thermal image reconstruction method. The reconstructed images are used to create derivative images, which are slopes extracted from the equation representing the reconstructed data $\ln(T) = \ln\left(\frac{Q}{e}\right) - \frac{1}{2}\ln(\partial t)$,

where *T* is the temperature of the sample, *Q* is the heat applied to the surface, *e* is the effusivity of the material, and *t* is time. Here, the slope is –1/2, which represents an area free of discontinuities. Deviations from this linear behavior can then be used as a discontinuity detection method and the time at which the deviation occurs as an indication of the discontinuity's depth. Establishing the deviation from linearity as the discontinuity detection criteria eliminates much of the subjectivity in the identification and characterization of subsurface discontinuities. Both first and second derivative images are derived in this way.

These derivative images should indicate discontinuities earlier than the temperature vs. time plots since the rate of cooling is more sensitive than the absolute measure of temperature (*12*).

The raw signal and the first and second derivatives are found at their peak contrast times with respect to the deepest discontinuity. Both derivatives significantly enhance the detection of the deeper feature, but the second derivative result is considerably stronger. It has been clearly shown that the derivative images are the most sensitive thermal parameters for detecting the discontinuities within a composite specimen. Utilizing the reconstructed data set is the most successful technique of data analysis. In addition, discontinuity depth can be calibrated based on the time at which deviation from linearity occurs (*11*).

With some subjective manipulation, pulsed thermography shows the discontinuities more clearly than either ultrasonic or radiographic methods (*11*).

The dynamic characterization technique is based on shifts in measured vibration frequencies and can directly relate vibration parameters to the anomalies in the structural stiffness or mass of ceramic composite specimens. It requires a mechanical force input into the structure as well as a nonbiased measurement of the vibration of the structure. Digital signal processing is necessary in order to clean up the signals and extract the vibration frequency components from the signals. When the specimens are continuous materials, multiple excitable natural vibration modes can be expected. For structural assessment, it is essential that the exact shift in each vibration frequency be determined through the identification of the various modes of vibration. This part of vibration testing is called mode identification. Access to the specimens themselves may not be possible. Hence, noncontact sensing techniques such as laser vibrometry are sometimes proposed.

The shift in natural vibration frequencies has been used as a quality indicator for likely manufacturing variables. Contact and noncontact results have been compared with theoretical natural frequency values and have shown that laser results are “noisier” due to dropout from speckle noises. It has been shown experimentally that dynamic characterization is a valid NDT technique for CMCs provided the manufactured parts have a quality variation greater than 3.21% (*12*).

4. Finite-Element Analysis and CMCs

The finite-element method is an approximation method for studying continuous physical systems used in structural mechanics, electrical field theory, and fluid mechanics. The system is broken into discrete elements interconnected at discrete node points, often regularly spaced into a so-called “grid” or “mesh” (*13*). The method can become very sophisticated mathematically, characterizing its solutions in such abstract mathematical spaces from functional analysis as $C^m(\Omega)$, a continuity class, and $H^m(\Omega)$, a Sobolev class. These spaces quantify the mathematical concept of "regularity" or degree of smoothness of a solution, the most important mathematical concept associated with the finite-element method (*14*).

A 3-D NDT (computer tomography) inspection of a SiC/SiC composite panel was carried out in addition to a finite-element analysis by Abdul-Aziz et al. (*15*). A CAD version of the tensile specimen was used for the 3-D finite-element model. Only half of the specimen was modeled due to symmetry. The finite-element model consisted of 1350 eight-node hex elements and 1932 nodes. Symmetry boundary conditions were applied to the center portion to simulate the rest of the specimen and suppress rigid body motion. The bottom corner node was restrained from moving along the z-plane, and tensile load along the axial direction was applied at the end section of the specimen to induce tensile loading conditions. These are all requirements of the finite-element method. In addition, the finite-element analyses were performed under linear elastic conditions, and a resulting stress output was generated, reporting a stress riser of 33% above the maximum stress to failure. The method predicts that a failure location will be initiated at these stress risers. Analytical results obtained showed that the high stress regions correlated well with the damage sites, as identified by the computed tomography scans and the experimental data. The combined NDT/finite-element analysis technique was able to quantify the effect of the observed voids in determining the likelihood of the failure sites in the tensile composite specimen, successfully capture the structural abnormalities of the CMCs, and identify the critical regions within the specimens.

5. Future Work

The 10 NDT methods described in this report will be further examined and prioritized as to their effectiveness in examining CMCs. Then the top three or four techniques will first be used to test for cracks, voids, porosity, and delaminations in the CMCs of importance to the U.S. Army. In addition, the finite-element method will continue to be combined with NDT to examine new CMCs in new ways. As new and more effective CMCs are developed, new and more effective techniques for inspecting and characterizing them will continue to be developed.

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